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(54) **Process for producing precision cartridge filter.**

(57) A process for producing a precision cartridge filter comprises the steps of :

(A) preparing a web composed of conjugate microfins fibers by a conjugate melt-blow process, which conjugate microfins fibers are composed of a lower melting point component and a higher melting point component having a melting point difference between them of 20°C or more ; and

(B) heating the web and molding it to the shape of a cartridge filter at a temperature at or about the lower melting point or higher but below the higher melting point, to obtain a cartridge filter having an average fiber diameter of 10  $\mu$ m or less.

This invention relates to a process for producing a precision, cylindrical cartridge filter, by subjecting a web composed of conjugate melt-blown microfine fibers to forming under heat into a shape of a cartridge filter.

A cartridge filter using melt-blown fibers (hereinafter often referred to as cartridge) is disclosed in Japanese patent application laid-open No. Sho 60-216818. Further, Japanese patent publication No. Sho 56-43139 discloses a process for producing a cylindrical fiber aggregate by after forming a web of conjugate short fibers by means of a carding machine, rolling up the web under heat. Still further, Japanese patent publication No. Sho 56-49605 discloses a process of inserting a sheet-form material having small pores into an intermediate portion of a cartridge.

However, the melt-blown fibers of the above Japanese patent application laid-open No. Sho 60-216818 are composed of a single component, and is a material having almost no bond of fibers to one another and retained by mechanical snarl or entanglement to one another and further having weak strength in the form of a cartridge filter; thus the fibers employ a structure provided with a central supporting member, so that filter production is not simple. Further, the fibers have a percentage of void as high as 80 to 90% so that it is difficult to retain the outer diameter of the cartridge filter even when it is used under low pressure. In particular, the smaller the particles to be filtered, the smaller the fiber diameter of the filter, so that notable shrinkage of the outer diameter of the filter occurs, resulting in anxious filtrability.

In the case of the cartridge using conjugate fibers, disclosed in the above Japanese patent publication No. Sho 56-43139, since short fibers cut to a definite length are processed, the fineness (denier) and the cut length of the fibers used have been naturally restricted. On the present level, stabilized carding of fibers of 1 d/f (denier/filament) or less is so difficult that it has been impossible to produce a cartridge capable of filtering off impurities of 10  $\mu$ m or less. In order to overcome such a drawback, the above Japanese patent publication No. Sho 56-49605 discloses a process of inserting a sheet-form material having small pores into an intermediate layer of a cartridge. However, the difference between the diameter of the fibers composing the sheet-form material and the diameter of the fiber of the cartridge body is so large that it is presumed that the layer of the sheet-form material works as a rate-determining step in a mechanism of surface layer filtration as in the case of membrane pleat filter. Further, such a sheet-form material is of a different kind of a stock from that of the stock constituting the cartridge, so that there have been problems of ply separation, etc. in the case of filtration of a high pressure and high viscosity fluid. Further, in the case of using short fibers, in order to improve the handling of the processing, an antistatic agent (oiling agent)

has so often been attached to the fibers. Thus, such a cartridge prepared from the fibers having the oiling agent, the oiling agent dissolves in a filtrate and bubbles at the initial stage of filtration, so that it is the present status that the cartridge can be expressly washed and used in the field of foods or in a precision filtration.

## SUMMARY OF THE INVENTION

The object of the present invention is to provide a process for producing a cartridge filter having a stabilized filtrability the pore diameter of which governing a filtration accuracy is not broadened by a filtration pressure, and needing no central supporting member and no oiling agent.

The present invention has the following constitutions:

(1) A process for producing a precision cartridge filter which process comprises the following steps of:

preparing a web composed of conjugate microfine fibers by a conjugate melt-blow process, which conjugate microfine fibers compose of a lower melting point component and a higher melting point component and a melting point difference between them is of 20°C or more; and

heating said web and molding it to a shape of a cartridge filter at a temperature of said lower melting point or higher, but lower than said higher melting point, to obtain a cartridge filter having an average fiber diameter of 10  $\mu$ m or less.

(2) A process for producing a precision cartridge filter as set forth in item (1), wherein said web is processed to a non-woven fabric in advance of said step of heating and molding.

(3) A process for producing a precision cartridge filter which process comprises the following steps:

preparing a web composed of conjugate microfine fibers by a conjugate melt-blow process, which conjugate microfine fibers compose of a lower melting point component and a higher melting point component and a melting point difference between them is of 20°C or more;

winding up said web on a core while heating said web at a temperature of said lower melting point or higher, but lower than said higher melting point, to form a cylindrical body of a cartridge filter on the core; and

cooling said cylindrical body to a room temperature to obtain a cartridge filter having an average fiber diameter of 10  $\mu$ m or less.

(4) A process for producing a precision cartridge filter which process comprises the following steps:

preparing a web composed of conjugate microfine fibers by a conjugate melt-blow pro-

cess, which conjugate microfibrils compose of a lower melting point component and a higher melting point component and a melting point difference between them is of 20°C or more;

winding up said web on a core to form a cylindrical body of a cartridge filter on the core;

heating said cylindrical body at a temperature of said lower melting point or higher, but lower than said higher melting point; and

cooling said cylindrical body to a room temperature to obtain a cartridge filter having an average fiber diameter of 10 µm or less.

(5) A process for producing a precision cartridge filter according to item (3) or (4), wherein said web is processed to a non-woven fabric in advance of said step of winding up.

(6) A process for producing a precision cartridge filter as set forth in item (1), (3) or (4), wherein the lower melting point component and the higher melting point component of said microfibril conjugate fibers are selected from a group of combinations of polyethylene/polypropylene, polyester/copolymerized polyester, polyethylene/polyester, and polypropylene/polyester.

(7) A process for producing a precision cartridge filter as set forth in item (1), (3) or (4), wherein said conjugate melt-blow process comprises steps of independently melting a lower melting point thermoplastic resin and a higher melting point thermoplastic resin, and subjecting them to conjugate spinning through a die for melt-blow, provided with spinning nozzles from the periphery of which a high temperature gas is blown at a high speed, and blowing the resulting conjugate fibers onto a collecting net conveyor with the high temperature gas to obtain a web of conjugate microfibrils.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The conjugate melt-blow process referred to in the present invention means a process of independently melting two kinds of thermoplastic resins having a lower melting point and a higher melting point, respectively, subjecting them to conjugate spinning through a die for melt-blow, provided with spinning nozzles from the periphery of which a high temperature gas is blown at a high speed, and blowing the resulting conjugate fibers onto a collecting net conveyor to obtain a web of microfibrils. As the die for conjugate fibers, those of sheath-and-core type, side by side type, etc. may be exemplified, and as the gas for melt-blow, air, inert gases, etc. usually at 0.5 to 5 kg/cm<sup>2</sup>, 400°C and in a flow quantity of 1 to 40 m<sup>3</sup>/min. are generally used. The distance between the die for melt-blow and the conveyor varies depending upon the melting point of the thermoplastic resins, the blow-

ing conditions of the gas, etc., but the distance is determined so as not to cause a mutual-adhesion of the fibers, and it is preferably about 30 to 80 cm.

The cross-sectional shape of the fibers may be circular shape, triangular shape; T-shape or a shape further provided with a hollow part in the above shapes.

Examples of the resin of the fibers of the present invention are combinations of polyamides, polyesters, polyvinylidene chloride, polyvinyl acetate, polystyrene, polyurethane elastomer, polyester elastomer, polypropylene, polyethylene, copolymerized polypropylene, etc. or the mixtures of the foregoing. The melting point difference between the lower melting point component and the higher melting point component is at least 20°C. If the melting point difference is lower than 20°C, the higher melting point component, too, softens or melts at the time of heating, the fiber shape collapses to form a film. If the cartridge melts to form a film and the pore collapses, this has a great influence upon the filtrability, such as reduction in the water-permeability. The melting point referred to herein is generally measured by means of a differential scanning calorimeter (DSC) in which the melting point appears in the form of endothermic peak. In the case of non-crystalline, low melting point copolymerized polyesters, etc., since the melting point does not always appear clearly, it may be substituted with softening point and which is measured by a differential thermal analysis (DTA). Examples of conjugate combinations are polyethylene/polypropylene, polyester/copolymerized polyester having a lower melting point, polyethylene/polyester, polypropylene/polyester etc., but the combinations are not limited to those.

As to heating for forming the cartridge, the web of microfibrils according to conjugate melt-blow process is heated to a temperature of the melting point of a lower melting point component or higher, but lower than a higher the melting point of a higher melting point component of the conjugate fibers. The web may be formed under heat into a cylindrical shape of a cartridge using a forming device provided with a conveyor, a heater and a winding-up means, or the web may be once wound up on a core, and heating the web on the core, followed by cooling the web to a room temperature, and drawing out the core to obtain a cylindrical shape of a cartridge. The web may be processed to form a non-woven fabric before the above processing. As for a heating process for the web (or the non-woven fabric), a heat emboss process, a heat calendar process, a hot air process, a supersonic process, a far infrared heating process, etc. are exemplified. In particular, according to the far infrared heating process, the web obtained by the conjugate melt-blow process is not disturbed to obtain small unevenness of the thickness, and uniform mutual adhesion is effected so that the filtrability is stabilized.

The fiber diameter of the cartridge filter obtained in the present invention is 10  $\mu\text{m}$  or less. If it exceeds 10  $\mu\text{m}$ , the filtration accuracy of the cartridge filter lowers and also the diameter of flowing-out particles increases. The average fiber diameter was determined by average value in a photograph of a scanning electron microscope inside the cartridge.

In the cartridge filter of the present invention, conjugate microfibrils according to melt-blow process are used and the higher melting point component of the conjugate fibers are three-dimensionally bonded to one another by means of the lower melting component of the conjugate fibers; hence as to the pore structure, the pores are not broadened even by variation of water pressure, etc. and a highly accurate filtration is stably effected; and the filtration accuracy was as high as 1  $\mu\text{m}$ , for example.

Further, the cartridge filter of the present invention is of a structure composed of the conjugate microfibrils made by a conjugate melt-blow process, tightly joined to one another without using another kind of stock such as sheet; hence a high pressure and high viscosity fluid is stably filtered. Further, since the lower melting point component of the fibers are melt-bonded to one another at the contacting points thereof to make the higher melting point fibers a stiff structure, a central supporting member is made unnecessary to simplify the preparation of the cartridge and thereby obtain a cheap cartridge.

Further, since the cartridge filter of the present invention is unnecessary to use an antistatic agent (oiling agent) for fiber processing, it can be used even in the food field and in the field of precision filtration, without washing.

Since the process for producing a cartridge filter of the present invention comprises spinning conjugate microfibrils according to conjugate melt-blow process and continuously carrying out spinning and forming, the production yield of the cartridge filter has been improved.

The present invention will be described in more detail by way of Examples, but it should not be constructed to be limited thereto. In addition, the measurement methods described in Examples were determined according to the following methods:

#### Filtration accuracy:

One cartridge is fixed to a housing and water is passed therethrough from a 30  $\ell$  water vessel by circulation by means of a pump. After the flow quantity was adjusted to 30  $\ell/\text{min}$ , 5g of a cake of carborundum (#4000) is added to the water vessel. One minute after the addition of the cake, 100cc of the filtered water is sampled and is filtered through a membrane filter (capable of collecting particles of 0.6  $\mu\text{m}$  or more), and the particle size of the cake collected on the membrane filter was measured by means of a particle size

distribution measurement device in which the number of particles of each particle diameter was counted, to render the maximum diameter of flowing-out particles as filtration accuracy.

#### Compressive strength:

One cartridge is fixed to a housing and water is passed therethrough from a 30  $\ell$  water vessel by circulation by means of a pump in a flow quantity set to 30  $\ell/\text{min}$ . Into the water vessel is added a material (20 g) having an average particle diameter of 12.9  $\mu\text{m}$  and a distribution wherein particles of 40  $\mu\text{m}$  or less occupy 99% of the total particles, obtained by grinding and classifying the lower layer earth of volcanic ash soil, followed by agitating it, carrying out circulating filtration and reading the inlet pressure and the exit pressure when the fluid inside the water vessel has become clear. Addition of the classified material of the volcanic soil and reading of the pressure difference at the time of the clarification are repeated, and the maximum pressure loss (difference between the inlet pressure and the exit pressure) at the time when the outer diameter of the cartridge has been deformed is measured to be rendered as the compressive strength.

#### Average fiber diameter:

The respective five portions of a web or the inside of a cartridge are sampled, and the respective portions are photographed by means of a scanning electron microscope. With each photograph, the fiber diameters of optional 20 ends of the fibers are measured, to obtain an average fiber diameter of the total 100 ends.

#### Example 1

A polypropylene having a melting point of 165°C and a melt flow rate (g/10min., 230°C) of 35 as a core component of a conjugate fiber and a linear low density polyethylene having a melting point of 122°C and a melt index (g/10min., 190°C) of 25 as a sheath component of the conjugate fiber were spun in a conjugate ratio of 50/50 and at 260°C/260°C, using a die for sheath-and-core type melt-blow, followed by introducing high pressure air to blow it onto a metal gauze conveyor, to obtain a microfibril web. This web had an average fiber diameter of 2.6  $\mu\text{m}$ , a basis weight of 49.0 g/m<sup>2</sup> and a specific volume of 25.2 cc/g. When this web was observed by means of a scanning electron microscope, melt-adhesion between the fibers was hardly observed, and the web was good without any roping or shot. This web was once wound up, followed by feeding it to a heating device provided with a conveyor and a far infrared heater, heating it at 145°C, winding up and forming on a metallic core of 30 mm in the outer diameter, cooling the resulting

central core to a room temperature, and drawing out the core to obtain a cylindrical cartridge of 60 mm in the outer diameter, 30 mm in the inner diameter and 250 mm long. The average fiber diameter inside this cartridge was 2.6  $\mu\text{m}$ . Further, the filtrability was measured. As a result, the filtration accuracy was 2.5  $\mu\text{m}$ , the compressive strength of the cartridge was 6.0  $\text{kg}/\text{cm}^2$ , and the shrinkage of the outer diameter was not observed until the deformation. Further, bubbling at the initial period of the filtration was not observed at all.

#### Example 2

A polyester having an intrinsic viscosity of 0.61 and a melting point of 252°C, as a higher melting point component, and the same linear low density polyethylene as used in Example 1, as a lower melting point component were spun in a conjugate ratio of 50/50, using a die for side-by-side type melt-blow, followed by collecting a microfine fiber web formed on a conveyor, and continuously feeding this web to the heating device and processing it in the same manner as in Example 1 to obtain a cylindrical cartridge having the same size as in Example 1. The web fed to the heating device had an average fiber diameter of 5.7  $\mu\text{m}$ , a basis weight of 51.0  $\text{g}/\text{m}^2$  and a specific volume of 28.1  $\text{cc}/\text{g}$ . The average fiber diameter inside the cartridge was 5.5  $\mu\text{m}$ . The filtrability was measured. As a result, bubbling at the initial period of water passing was not observed at all. The filtration accuracy was 4.5  $\mu\text{m}$ , the compressive strength of the cartridge was 7.5  $\text{kg}/\text{cm}^2$  and the shrinkage of the outer diameter was not observed until the deformation.

#### Comparative examples 1 and 2

Two kinds of cartridge composed of non-conjugate melt-blown fibers of polypropylene, alone and provided with a central supporting member, were evaluated according to the same measurement method as described above. One of the cartridge samples have (1) a designation accuracy of 0.5  $\mu\text{m}$  (fiber diameter: 0.9  $\mu\text{m}$ ) and the other sample has a designation accuracy of 1  $\mu\text{m}$  (fiber diameter: 1.2  $\mu\text{m}$ ). As a result, the values of the filtration accuracy scattered at each measurement, but their average values were (1) 5  $\mu\text{m}$  and (2) 9  $\mu\text{m}$ , respectively. Further, the compressive strength was as low as 1.8  $\text{kg}/\text{cm}^2$  and also the shrinkage of the outer diameter was notable.

#### Example 3

In Example 2, a polypropylene having a melting point of 162°C and a melt flow rate ( $\text{g}/10\text{min.}$ , 230°C) of 85 as a first component (a higher melting point component) and a polyethylene having a melting point of 122°C and a melt index ( $\text{g}/10\text{min.}$ , 190°C) of 48 as a

second component (a lower melting point component) were spun using the same die as in Example 2, at 260°C/260°C, in a conjugate ratio of 50/50, followed by blowing the resulting fibers onto a metal gauze by high pressure air at 360°C, to obtain a microfine fiber web. This web had an average fiber diameter of 0.7  $\mu\text{m}$ , a basis weight of 49.0  $\text{g}/\text{m}^2$  and a specific volume of 29.7  $\text{cc}/\text{g}$ . This web was heated to 140°C and processed in the same manner as in Example 2 to prepare a cylindrical cartridge having an outer diameter of 60 mm, an inner diameter of 30 mm and a length of 250 mm. This cartridge had an average fiber diameter of 0.7  $\mu\text{m}$ . Further, its filtrability was evaluated to give a filtration accuracy of 0.8  $\mu\text{m}$ . The compressive strength was 6.5  $\text{kg}/\text{cm}^2$ , and the outer shape shrinkage was not observed until deformation. Further, bubbling at the initial period of filtration was not observed at all.

#### Example 4

In Example 1, before the web blown onto the metal gauze conveyor was sent to the heat treatment device, it was once treated by a calender roll at 110°C and a linear pressure of 8  $\text{kg}/\text{cm}$  to obtain a non-woven fabric.

This non-woven fabric was heated to 140°C using the same device as in Example 1 to prepare a cylindrical cartridge having an outer diameter of 60 mm, an inner diameter of 30 mm and a length of 250 mm. This cartridge had an average fiber diameter of 2.6  $\mu\text{m}$ . Further, its filtrability was evaluated to give a filtration accuracy of 2.4  $\mu\text{m}$ . Further, the compressive strength was 7.7  $\text{kg}/\text{cm}^2$ , and the outer shape shrinkage was not observed. Further, bubbling at the initial period of filtration was not observed at all.

#### Example 5

A microfine fiber web was obtained using the same device and processed in the same manner as in Example 1, except that a polyester having an intrinsic viscosity of 0.60 and a melting point of 252°C, as a core component, and a lower melting point, copolymerized polyester composed mainly of terephthalic acid, isophthalic acid and ethylene glycol and having an intrinsic viscosity of 0.58 and a melting point of 160°C, as a sheath component, were spun at 285°C/270°C blowing a high pressure air of 360°C. The web had an average fiber diameter of 1.8  $\mu\text{m}$ , a basis weight of 49.5  $\text{g}/\text{m}^2$  and a specific volume of 23  $\text{cc}/\text{g}$ . This web was observed by a scanning electron microscope. As a result, adhesion between the fibers was hardly observed, and the web was good without any roping or shot. The web was once wound up and heated to 200°C using the same device and processed in the same manner as in Example 1 to prepare a cylindrical cartridge having the same size as in

Example 1. This cartridge had an average fiber diameter of 1.9  $\mu\text{m}$ . Further, its filtrability was evaluated to give a filtration accuracy of 1.4  $\mu\text{m}$ . Further, it had a compressive strength of 7.5 kg/cm<sup>2</sup> and the outer shape shrinkage was not observed until its deformation. Further, bubbling at the initial period of filtration was not observed at all.

#### Claims

1. A process for producing a precision cartridge filter which comprises the steps of:

(A) preparing a web composed of conjugate microfines fibers by a conjugate melt-blow process, which conjugate microfines fibers are composed of a lower melting point component and a higher melting point component having a melting point difference between them of 20°C or more; and

(B) heating the web and molding it to the shape of a cartridge filter at a temperature at or about the lower melting point or higher but below the higher melting point, to obtain a cartridge filter having an average fiber diameter of 10  $\mu\text{m}$  or less.

2. A process according to claim 1, wherein the web is processed to a non-woven fabric before the heating and molding step (B).

3. A process according to claim 1 or claim 2 wherein the lower melting point component and the higher melting point component of the conjugate microfines fibers are selected from the combinations polyethylene/propylene, polyester/copolymerized polyester, polyethylene/polyester, or polypropylene/polyester.

4. A process according to any one of the preceding claims wherein the conjugate melt-blow process comprises the steps of independently melting a lower melting point thermoplastic resin and a higher melting point thermoplastic resin, and subjecting them to conjugate spinning through a melt-blow die, provided with spinning nozzles from the periphery of which a high temperature gas is blown at a high speed, and blowing the resulting conjugate fibers onto a collecting net conveyor with the high temperature gas to obtain a web of conjugate microfines fibers.

5. A process according to any one of the preceding claims wherein the heating of the web is carried out by a far infrared heating process, a hot air process, a supersonic process, a heat calender process or a hot emboss process.

6. A process according to any one of the preceding claims in which step (B) is effected by winding up the web onto a core while heating the web at a temperature at or above the lower melting point but below the higher melting point, to form a cylindrical body of a cartridge filter on the core; and cooling the cylindrical body to ambient temperature to obtain a cartridge filter having an average fiber diameter of 10  $\mu\text{m}$  or less.

7. A process according to any one of claims 1-5 in which step (B) is effected by winding up the web onto a core to form a cylindrical body of a cartridge filter on the core; heating the cylindrical body at a temperature at or above the lower melting point or higher but below the higher melting point; and cooling the cylindrical body to ambient temperature to obtain a cartridge filter having an average fiber diameter of 10  $\mu\text{m}$  or less.



European Patent  
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## EUROPEAN SEARCH REPORT

Application Number

EP 91 30 5974

| DOCUMENTS CONSIDERED TO BE RELEVANT  |   |  |   |
|--|---|--|---|
| Category   | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim                              | CLASSIFICATION OF THE APPLICATION (Int. Cl.5) |
| A  | US-A-4 443 513 (G.H. MEITNER)<br>---  |  | B 01 D 39/16<br>D 04 H 1/54                   |
| A  | EP-A-0 292 294 (CHISSO CORP.)<br>---  |  |   |
| A  | EP-A-0 171 806 (CHICOPEE)<br>---  |  |   |
| A  | EP-A-0 105 729 (CHICOPEE)<br>-----  |  |   |
|  |   |  | TECHNICAL FIELDS<br>SEARCHED (Int. Cl.5)      |
|  |   |  | B 01 D 39/00<br>D 04 H 1/00                   |
| The present search report has been drawn up for all claims   |   |  |   |
| Place of search<br>THE HAGUE   |   | Date of completion of the search<br>15-10-1991 | Examiner<br>DEVISME F.R.                      |
| <p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone<br/> Y : particularly relevant if combined with another document of the same category<br/> A : technological background<br/> O : non-written disclosure<br/> P : intermediate document</p> <p>T : theory or principle underlying the invention<br/> E : earlier patent document, but published on, or after the filing date<br/> D : document cited in the application<br/> L : document cited for other reasons<br/> &amp; : member of the same patent family, corresponding document</p> |   |  |   |

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